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AN EFFECT OF STRESS RELIEVING ON THE MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF Mg-3Zn-1Cu-0.7Mn/ Al_2O_3 COMPOSITE SYNTHESIZED BY POWDER METALLURGY

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ABSTRACT

A composite based on Mg-3Zn-1Cu-0.7Mn alloy reinforced with Al_2O_3 was synthesized using blend-press-sinter powder metallurgy route and hot extruded at 400°C. The study concentrates on investigating the effectiveness of alumina as reinforcement and analyzing the effect of stress relieving on the mechanical and microstructural properties of the composite. The results of the study indicate that the ultimate tensile strength, yield strength and fracture strain of the composite was increased in the composite compared to the pure Mg metal. It is also proved that stress relieving has a significant role on the ultimate tensile strength and fracture strain in the composite.

KEYWORDS: Mg-Zn Composites, Powder Metallurgy, Stress Relieving, Tensile Strength, Yield Strength, Fracture Strain & Microstructure

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Original Article

INTRODUCTION

As magnesium-based materials display superior mechanical properties and show better strength to weight ratio, plenty of research activities are going on in this area [1-4]. There is a huge demand for modern materials with the development of science and technology as well as the grown demand for the latest technology by human beings. The conventional metallic alloys, polymers, ceramics, etc fail in satisfying the advancing human needs. This scenario promotes the requirement and development of new composites which render a variety of properties with lightweight [4,5].

The combination of metal matrix and different reinforcements represent metal matrix composites (MMCs). The metal matrix and nature of reinforcement is selected based on the nature of the properties expected for the composite. The last two decades witnessed abundant studies related to the effect of different types of reinforcements on metal matrix composites [6-9]. Based on the processing route, matrix composites exhibit a variety of properties. Solid phase processes are commonly used for the manufacture of composites with fine microstructural features, which display enhanced strength properties. Powder metallurgy is one of the simplest modes of solid phase process to synthesize MMCs [6-8].

The most commonly used metal matrix materials are pure metals or conventional metallic alloys. There are several alloys based on magnesium and aluminium used as metal matrix materials. Among the Mg-based alloys, Mg-Al and Mg-Zn series are generally used. As far as Mg-Al alloys are concerned, the Al content in the alloy boosts the grain refinement and provide good yield strength and ultimate strength at room temperature. Compared to Mg-Al series alloys, precipitation strengthening can easily be attained in Mg-Zn series alloys. It is reported that Mg-Zn series alloys are more stable at room temperature than the other. Also, Mg-Al series displays good creep resistance [10,11].

The properties of composites depend upon certain parameters, like the route in which the material is processed, the metal matrix used and the nature of the reinforcement. The mechanical properties can further be altered by secondary processing and heat treatment [6,12]. Therefore the selection of suitable matrix-reinforcement combination and the processing route are critical for procuring the optimum properties desired.

The elemental form of magnesium lacks in mechanical properties, as low elastic modulus, limited ductility and poor creep resistance. This scenario can be overridden by introducing magnesium-based alloy composites reinforced with ceramics as SiC and Al_2O_3 particles [13-17]. It is reported that when the Mg metal matrix is reinforced with Al_2O_3, excellent mechanical properties are obtained, compared to the elemental magnesium metal. The properties improved belong to the microhardness, strength, and ductility of the MMC [18,19].

This study focuses on fabricating a cheap composite based on Magnesium alloy with Al_2O_3 as reinforcement and investigating the role of stress relieving on the mechanical properties of the composite.

EXPERIMENTAL PROCEDURE

A composite billet of Mg-3Zn-1Cu-0.7Mn/ Al_2O_3 is fabricated with elemental metallic powders of Mg (99.99 wt.% purity, 200 mesh), Mn (99.99 wt.% purity, 200 mesh), Cu (99.99 wt.% purity, 200 mesh), Zn (99.99 wt.% purity, 200 mesh) and Al_2O_3 as reinforcement. The processing route adopted for the manufacturing of the composite is a blend-press-sinter powder metallurgy process. The homogeneous mixing of the metal powders and reinforcement is performed during the blending process using a ball milling machine maintained at a ball feed ratio of 3:1 and at 100 rpm for 24 hours. The mixture is compacted in a die of diameter 65 mm applying 1500 kg load with a hydraulic power press to get the composite billet and sintering is executed in the argon atmosphere at 400^{o}C for 1 hour in an automated electronic muffle furnace. The sintered billet is extruded in a die with a 5.4:1 ratio using a hydraulic power press operated at 0.2 to 0.5 mm/s speed and a 2000 kg load at 400^{o}C.

The automated electronic muffle furnace is again utilized to stress relieve the extruded material at 260^{o}C for a duration of 15 minutes in an argon atmosphere. The properties of the composite samples are analyzed with two categories, one without the stress relieving i. e., as-extruded (represented as F1) and the other with stress relieving (represented as F2).

ASTM standard of E8M-16a is followed for the preparation of tensile specimen and the test is performed at a strain rate of $1 \times 10^{-3}\ s^{-1}$. The optical microstructure is captured with etched specimen using the etchant blended with 1.5 g picric acid, 25 ml ethanol, 10 ml acetic acid, and 10 ml distilled water. The detailed phase analysis is accomplished by X-ray diffraction (XRD) and the intermetallic phases across the microstructure involved are found out. A scanning electron microscope (SEM) is utilized to capture the fractographic images of the cracked specimen after the tensile test.

RESULTS AND DISCUSSIONS

Microstructural Analysis

The microstructural analysis of the samples prepared according to the ASME standard of E8M-16a reveals the morphological changes that happened during the manufacturing process. Figure (1) represents the optical microstructure of the polished etched composite specimens F1 and F2. The microstructural analysis indicates the uniform distribution of Al_2O_3 particle across the entire microstructure with minimum micropores, which shows the bonding that occurred in between the metal matrix and reinforcement. This may be due to blending parameters used for homogeneous mixing and due to the higher extrusion ratio used. Even though there is a difference between the particle sizes of matrix and reinforcement, a high deformation level in extrusion caused a uniform distribution of Al_2O_3 across the matrix. The bonding between the matrix and reinforcement may be due to the limited presence of microvoids and the interfacial reaction between the matrix and the reinforcement [19-21]. The grains are observed as well refined due to the complete recrystallization that happened during the hot extrusion process and near equiaxed grains are seen in the micrograph of the specimen after the stress relieving process (F2) compared to the other (F1). The size of the equiaxed grains in the stress-relieved composite is observed to be bigger compared to the as-extruded composite. The grain refinement took place may be primarily due to the ability of Al_2O_3 particles to nucleate Mg grains during the recrystallization process. The pinning effect of Al_2O_3 particulates on Mg grains that caused for restricted grain growth during the recrystallization process. It may also be a reason for the grain refinement.

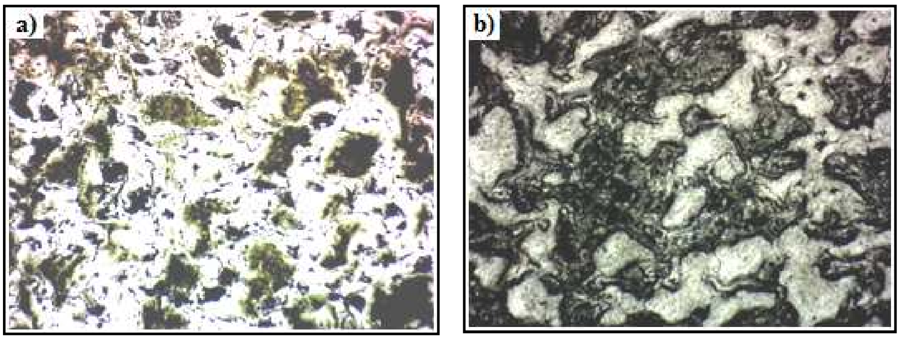

Figure 1: Optical Micrographs of the Extruded ZC31M/ Al_2O_3 Composites Intered at 400°C (a) As Extruded Alloy Composite (F1) (b) Stress Relieved Alloy Composite at 260°C (F2)

The study reveals the following parameters, (a) distribution of reinforcement across the entire microstructure (b) the bonding between the alloy matrix and the reinforcement (c) minimum porosity of the composite and (d) the interfacial characteristics of matrix and reinforcement.

Density and Porosity Results

The theoretical density, actual density, and porosity are given in Table 1. The porosity calculated is minimum as the value of theoretical density and actual density is very closer. However, there is a significant reduction in porosity of the composite after the stress relieving process. This may due to the following reasons: 1) There may be trapped gases inside the sample during the compaction process forming microvoids. It may be released during the stress relieving. 2) The evolution of hydrogen may also result in increased porosity [22-25]. The minimum porosity values imply a blend-press-sinter powder metallurgy process is a successful method for the manufacture of metal matrix composites.

Table 1: Density and Porosity of the Composite Samples at Room Temperature

Sl. No	Material	Theoretical Density (g/cc)	Experimental Density (g/cc)	Porosity (%)
1	Mg-3Zn-1Cu-0.7Mn/Al_2O_3composite (F1)	1.834	1.825 ± 0.003	0.49 ± 0.006
2	Mg-3Zn-1Cu-0.7Mn/Al_2O_3composite (F2)	1.834	1.829 ± 0.003	0.27 ± 0.005

X-ray Diffraction Studies

The different phases involved in the microstructure are recognized with XRD analysis conducted on the polished alloy composite specimen employing the Cu-Kα radiation of λ = 1.5406 Å at a scanning speed of 2^o/min, an applied voltage of 40 kV and beam current of 30mA. The Bragg angle and lattice spacing are compared with the standard values of the related phases of magnesium, Al_2O_3, copper, zinc, and manganese [26]. Figure (2) depicts the diffractogram obtained from the XRD analysis. It gives the different intermetallic phases associated in the microstructure of the alloy composites which comprise the phases of Mg and Al_2O_3, Cu_5Zn_8, MgZnCu, and $Al_{0.93}Cu_{1.07}Mg$. The XRD analysis authenticates the different phases dispersed within the matrix.

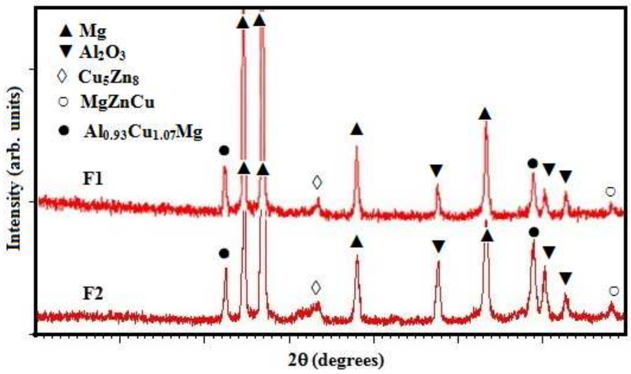

Figure 2: X-ray Diffraction Patterns for as Extruded ZC31M / Al_2O_3 Composite (F1) and Stress Relieved Alloy Composite at 260°C (F2) Sintered at 400°C

Mechanical Properties

The mechanical properties of the composite samples such as ultimate tensile strength, yield strength, and fracture strain are given in table 2. The highest ultimate tensile strength is achieved for the stress relived composite (F2). But the yield strength is reported to be higher for the as-extruded composite (F1) compared to the stress relieved composite (F2). A fracture strain of 6.88% is shown by the stress relieving composite which is more than the as-extruded composite. All the mechanical properties are superior when compared with the pure magnesium metal. The engineering stress-strain curve for the composite samples is given in figure 3.

Table 2: Tensile Properties of the Composite Samples at Room Temperature

Material	Ultimate Tensile Strength UTS (MPa)	Yield Strength YS (MPa)	Fracture Strain (%)
Pure Mg [22]	150	105	5
As-extruded composite (F1)	221.41	144.15	5.98
Stress relieved composite (F2)	223.62	129.44	6.88

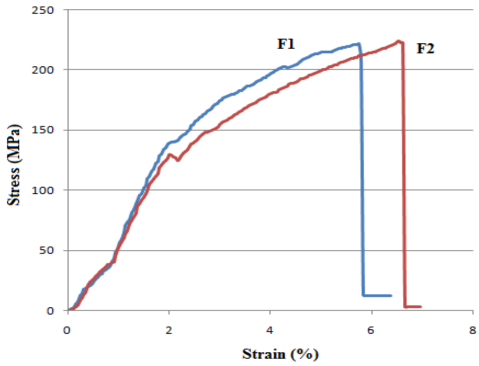

Figure 3: Engineering Stress-Strain Curve of Composite Specimens (a) As-Extruded (F1) (b) Stress Relieved (F2)

The improvement in mechanical properties of Mg-based materials is governed by several mechanisms, which are explained below: i) Well refined grains are obtained when the Mg matrix is reinforced with ceramic particles, which in turn increases the area of grain boundary and enhances the strength of the material. ii) When the reinforced phase is distributed uniformly across the magnesium matrix, the surface integrity between the matrix phase and the reinforcement phase increases which magnifies the strength of the material. iii) Another parameter that influences the strength of composites is the movement of dislocation. The dislocation movement is stopped or hindered by the ceramic oxide particles which lead to the accumulation of dislocations and increases the strength of the composite. Yield properties are heavily influenced by the dislocation movement. iv) The difference in CTE of the matrix and reinforcement creates thermal stress to induce at elevated temperatures in between the matrix and reinforcement interface. The direction of thermal stress may be in any direction and it forms multi-glide planes along which the dislocations move. But when the material is loaded, thermal stresses are evolved in multiple directions and forms multiple glide planes. The accumulation of multiple glide planes produces ledges at the grain boundary which prevents the free movement of dislocation and increases the strength of the materials.

The mechanical properties particularly the UTS and fracture strain are found to be better for the stress relieved alloy. This may be due to the reduced number and size of micropores present in the composite specimen. The stress relieving process reduces the number and size of the micropores which improves the mechanical properties here.

The figure 4 shows the fracture surfaces of the composite after the tensile test which is comprised of a lot of dimples and ridges. This is the evidence for the ductile fracture that occurred during the fracture of the specimen while undergoing the tensile test. The dimples that are visible in the figure 4(a) is shallower compared to the figure 4(b).

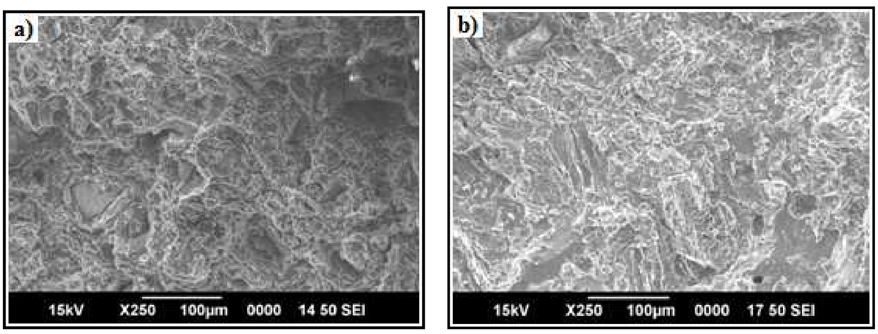

Figure 4: SEM Images of the Tensile Fractographs of (a) As Extruded Alloy (F1) (b) Stress Relieved Alloy (F2)

The deeper dimples are proof for the improved fracture strain in the stress-relieved alloy. This may due to a lesser presence of micropores in the composite after the stress relieving process. The small phases visible in both the fractographs cause for the stress concentration and act as the center of stress concentration. The cracks are originated from this center of stress concentration. As microstructure of the stress relieved alloy is homogeneous and lesser number voids are present in the microstructure, the evolution of crack is propagated very slowly. It may the reason for the improved ultimate tensile strength and fracture strain in the stress-relieved alloy.

CONCLUSIONS

A composite of Mg-3Zn-1Cu-0.7Mn/Al2O3 is synthesized using blend-press-sinter powder metallurgy and hot extruded for enhancing the mechanical properties. The study focused on analyzing the effect of alumina as reinforcement on the Mg alloy matrix composite and the effect of stress relieving on the ultimate tensile strength of the composite and investigating the mechanisms associated with it. It may be concluded from the study as follows.

- From the study, the ultimate strength, yield strength and fracture strain of the composite synthesized are found to superior compared to that of the pure Mg metal and the effect of alumina as the reinforcement on the mechanical properties of alloy matrix is successfully proved. This may be due to the uniform distribution of the Al_2O_3 reinforcement phase in the alloy matrix and the surface integrity between the primary and secondary phases. The XRD analysis revealed the different phases involved in the composite. The ledges at the grain boundary formed by the accumulation of multiple glide planes also caused for the superior strength of the composite by restricting the dislocation movement.
- When the composite Mg-3Zn-1Cu-0.7Mn/Al_2O_3 is stress relieved at 260^o, it is observed that the stress relieving process has a significant influence on the mechanical properties of the composite. The ultimate tensile strength and fracture strain improved after stress relieving compared to that of the as-extruded composite as the size and number of micro pores is reduced during the stress relieving process which is proved in the optical microscopic analysis.
- The fracture analysis of the tensile specimen revealed that ductile fracture has transpired during the tensile test. The small intermetallic phases portrayed as the center of stress concentration and the ledges at the grain boundaries and the small phases slowed the propagation of cracks and caused for the improved strength of the composite.

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